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A PILOT PLANT FOR SOLVENT EXTRACTION STUDIES[1]

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Peoria, Illinois

In the solvent extraction of soybeans, usually eight different operations take place. These can be considered in two groups--soybean preparation and soybean extraction. All of these individual operations affect the economy of the process and the quality of the products.

The equipment at the Northern Regional Research Laboratory is designed to process soybeans by a continuous operation at the rate of 30 pounds an hour. Although this capacity is small, yet practically all the problems encountered in commercial units are present in the pilot plant, and its design is such that operating conditions can be changed and research data taken on many of the operations. This description of the pilot plant starts with the first step in the milling operations and continues with the succeeding operations in their regular order.

The efficiency of the extraction process depends to a great extent upon the quality and condition of the flakes. First, the beans are cleaned of foreign material and elevated to the storage bin of the pilot plant. This bin has a capacity of 15 bushels and it, together with other equipment used for preparation of the flakes, is indicated on the accompanying flowsheet, Figure 1. From the storage bin, the beans move to the cracking rolls through a variable-speed screw conveyor, the speed of which can be regulated to give the desired feed rate. The beans are cracked in a set of corrugated rolls, 6 inches in diameter, which turn at different speeds. These rolls crack the beans to particles ranging in size from eighths to quarters. It is highly desirable to produce uniformly cracked particles with a minimum of fines.

The cracked beans are then conveyed to the dehulling equipment. This consists of a shaker screen with air aspiration to remove loose hulls and a scourer which removes the hulls that did not break free previously. The dehulling equipment is used only for the production of extracted flakes which are suitable for edible use or for other special purposes. Normally, the dehulling apparatus is bypassed and the cracked beans are conveyed directly to the tempering conveyor.

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[1] Revised from material originally presented by R. H. Gustafsson at the Cooperative Soybean Oil Mills Conference at the Northern Regional Research Laboratory, Peoria, Illinois, May 25-27, 1948.

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The temperer is a paddle conveyor provided with a hot water jacket and with means for adding steam directly for moistening the beans, if necessary. This equipment heats and adjusts the moisture content of the cracked beans, as required, in order to produce good flakes. In commercial practice, the conditions for tempering the beans vary somewhat among plants; the amount of moisture desired and the temperature to which the beans are heated depend on the condition of the whole beans and on the type of flaking rolls in use. Processors strive to produce flakes that are thin but uniform in thickness, not easily broken, and which have a definite bulk density. Very little information has been published concerning the best manner for tempering the beans in order to produce the proper type of flakes. It may be that certain temperatures moisture contents, and times for distribution of the moisture are more beneficial than others.

In the pilot-plant operations at this Laboratory, the beans are heated to about 160° F., and the moisture content is adjusted to about 10 percent. The flakes are prepared by a set of smooth rolls 12 inches in diameter. These flakes are about the size of a fingernail and 0.008 inch thick. Uniformity of thickness is highly desirable for regularity in the extraction process. The flakes are made as thin as is practicable, however, since this reduces the time required for the extraction of the oil. From experimental studies in the pilot plant, it has been learned that more than 90 percent of the oil is removed from the flakes within the first few minutes, but extraction of the remainder requires as much as an hour's retention time. The amount of fines and the friability of the flakes are important factors which affect the flow of the solvent. It is desirable to retard the solvent somewhat in its flow through the flakes, but fines may cause the solvent to seek the path of least resistance so that channeling occurs, with a resultant drop in extraction efficiency. Also, a varying fraction of the fines leave with the miscella. They must be removed by filtration or other means and, when they are added to the extracted residue, they lower the overall extraction efficiency because of their high-oil content.

In small flaking rolls, the problem of producing good flakes is always present, and many of the troubles in the pilot plant do not occur in commercial plants since they use larger rolls, some as large as 4 feet in diameter. Large rolls nip the cracked beans readily. As the size of the rolls is diminished, their ability to nip the beans decreases. Consequently, at high feed rates, material may accumulate on top of the rolls.

The flaked beans must be dried when 95 percent ethyl alcohol is to be used as the solvent, but when hexane is to be employed the flakes may be sent directly to the extractor. If the flakes contain 3 percent or less of moisture, the alcohol will not dissolve any of the water and the solvent may even be partially dehydrated. The drier used at the Northern Laboratory is one designed and constructed by the Laboratory staff. It is essentially an endless screen belt which supports the flakes while heated air passes up through the screen and flake bed. The versatility of the equipment is well illustrated by this flake drier. A flake bed of variable thicknesses can be spread on the belt, any retention time from 5 to 30 minutes may be selected, the amount of heated air passing through the flake bed can be adjusted, and the temperature of the heated air can be controlled. If it should be desirable, it would even be possible to dry the flakes in an inert atmosphere. In addition, all of the parts are simple in construction and accessible for adjustment.

The milling equipment where the beans are prepared for extraction includes the bean storage hopper, the variable-speed feeder conveyor, the cracking rolls, the dehulling equipment, the temperer, and the flaking rolls. The various pieces of equipment are connected by bucket-type elevators and screw conveyors to provide for continuous flow of the material. These elevators and conveyors are located back of the equipment. The arrangement would have been simpler, however, if the equipment could have been located so as to use gravity flow from one unit to the next.

One of the biggest problems in solvent extraction is material flow. Moist soybean flakes are capable of forming solid plugs in conveyors, rotary valves, and even in the larger pieces of equipment. All conveyors, elevators, and rotary valves should be made as self-cleaning as possible. Also, if wet beans or flakes are allowed to accumulate anywhere in the sytem, their decomposition will produce a very obnoxious odor and, of course, the decayed matter will contaminate the product.

The two processes of milling and extraction are carried out separately in different rooms with exhaust fans for each. The electrical equipment and wiring in the milling room conform to the National Electrical Code for Class II, Group G, hazardous locations; and those in the extraction room comply with Class I, Group D, requirements.

Figure 2 is the flowsheet for the extraction plant. The Kennedy-type extractor in which the flakes are processed is about 25 feet long and 10 inches wide. It has 20 stages or sections, each one of which consists of a sump and a paddlewheel. The wheels rotate very slowly at a rate of approximately one revolution every 5 minutes, and the flakes are pushed very gently through the solvent from one stage to the next. Each section is provided with an outlet through which samples of the miscella can be withdrawn, the miscella being a solution of the solvent and the oil. The extractor is placed on a slight slope so that the solvent which is introduced at one end flows by gravity, countercurrent to the flakes, to the other end of the extractor. The flakes are fed into the extractor by a screw conveyor, the speed of which can be varied. The drive for the paddlewheels can be adjusted to give any desired retention time ranging from 15 minutes to 2 hours. The temperature of the process can be controlled by regulating the temperature of the water in a jacket beneath the extractor. The solvent is preheated by a small steam heater, and the desired flow rate is obtained by use of a proportioning pump. The usual retention time for soybean flakes having a thickness of 0.008 inch is

1 hour, and a ratio of one pound of solvent per pound of flakes is employed. This generally produces a marc having a residual oil conent of 0.8 percent. With only slight deviations, these conditions are typical of those used in some commercial plants.

Of course it is desirable that the ratio of solvent to beans be as low as possible without detracting from the efficiency of the extraction. With lower ratios there is less solvent to be removed from the oil in the evaporation step and, consequently, less steam and cooling water are required.

After the flakes pass through the last section of the extractor, they are elevated by a drag-link conveyor which allows the excess solvent to drain away. They enter the flake desolventizer through a rotary valve which acts as a vapor seal. At this point the extracted residue contains approximately 35 percent of moisture and volatile matter. The desolventizer is designed to evaporate the solvent from the flakes by the use of low-pressure steam. Removal of the solvent at low temperatures and in the absence of open steam is desirable when the protein in the flakes is to be utilized for some special purpose other than for feeds. The construction of the desolventizer is somewhat similar to a French stack cooker. The unit used at the Northern Laboratory consists of four steam-heated shelves placed one above the other with revolving scrapers that move the material across a shelf until it falls through an annular opening to the shelf beneath. The desolventized flakes are removed by a screw conveyor and pass through another rotary valve which serves as a vapor seal. These valves are constructed with spring-loaded vanes to provide for taking up the wear. Open steam is used to strip the last traces of the solvent from the meal.

The vapors leaving the flake desolventizer are filtered to remove any dust which may foul the condenser tubes and also help to form an emulsion in the water-solvent separator. The dust filter is steam-jacketed to prevent condensation of solvent vapors as they are filtered through a layer of flannel cloth. The vapors are then condensed in an ordinary tube-and-shell condenser. The condensate passes through a solvent-water separator before the solvent is returned to the storage tanks and the water is discharged to the sewer.

The miscella coming from the extractor is collected in storage tanks and then filtered in an enclosed, horizontal plate type filter to remove the fines. The solvent and miscella storage tanks are mounted on scales so that readings can be made on a weight basis rather than a volumetric basis. The filtered solution is pumped to intermediate tanks and fed to the solvent removal system. The solvent is removed from the oil in two steps. The major portion is recovered in an evaporator of the natural recirculation type and the remainder of the solvent is separated from the oil in a stripping column. The flow rate of miscella to the evaporator is measured by a rotameter. The evaporator consists of a verticle tube bundle which contains 7 tubes, each of which has a diameter of 3/4 inch and a length of 5 feet, a vapor-liquid separator, a recirculation line, a liquid-level controller, and a preheater for the miscella feed. The evaporator is operated under a vacuum of 20 inches of mercury with steam at gauge pressures of 2 to 20 p.s.i. in the shell. Regulators are provided for controlling the vacuum in both the evaporator and the stripping column.

The oil solution, which should be concentrated to at least 90 percent in the evaporator, is stored in intermediate tanks and drawn from there through a rotameter and preheater into the stripping column. This stripping column is fabricated from a vertical tube bundle with seven tubes, each of which is 8 feet long with an outside diameter of 1 inch, beneath which is a section of pipe filled with 1/2-inch Raschig rings to a depth of 18 inches, an oil reservoir, a distributor at the upper end of the tube bundle for the feed introduced at that point, and an entrainment separator. Superheated steam is introduced beneath the packing to aid in the removal of the solvent from the oil. The column is operated under a vacuum of 20-22 inches of mercury with steam at a gauge pressure of 15 pounds per square inch in the shell. The crude oil leaving the stripping column passes through a cooler and is collected in a receiver.

The process flow described above is that used when hexane is employed as the extraction solvent. When ethyl alcohol is used, some modifications must be made in handling the miscella. Instead of the evaporator, a miscella cooler and a phase separator are employed. The mixture of ethyl alcohol and soybean oil is cooled in a heat exchanger from 172° F. to 70° F., or lower when necessary, and then it is fed into a tank with a conical bottom which serves as a phase separator. An alcohol layer containing about two percent of oil and two and one-half percent of other non-volatile constituents accumulates in the phase separator along with an oil layer containing about seven percent of solvent, and a layer of non-oil solids that come out of solution. The alcohol layer is reheated and returned directly to an intermediate point in the extractor. The oil layer in the phase separator is processed in the stripping column. Open steam cannot be used in the flake desolventizer and stripping column in this case, as the recovered alcohol would be diluted. The oil-free alcohol is introduced at the conventional solvent inlet in the extractor.

This pilot plant is being employed for investigations related to the solvent extraction of safflower seed, the use of ethyl alcohol and other solvents for the extraction of oilseeds, the improvement in the quality of soybean meal as a feed for livestock, and for the preparation of materials of known history for other research groups in the laboratory.

FIG. 1.– OILSEED MILLING

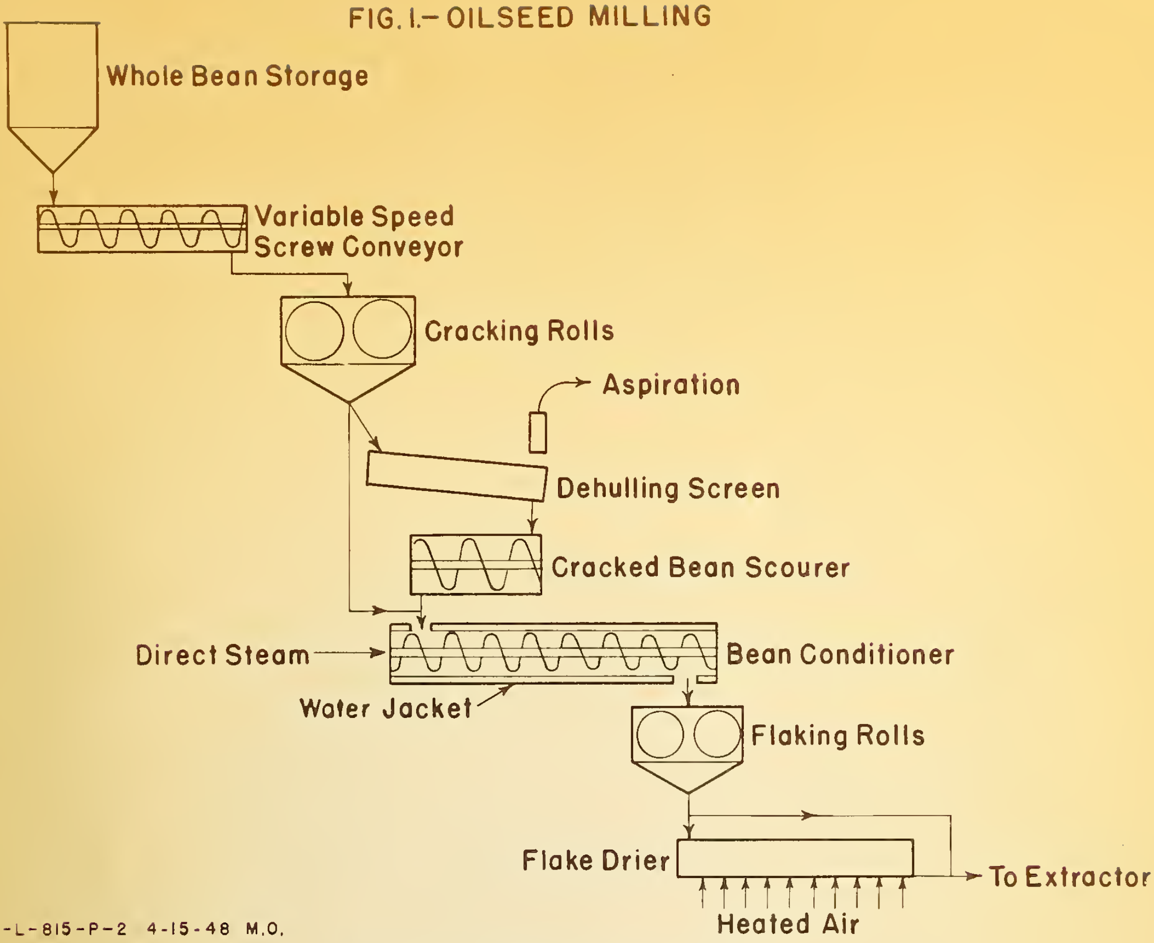

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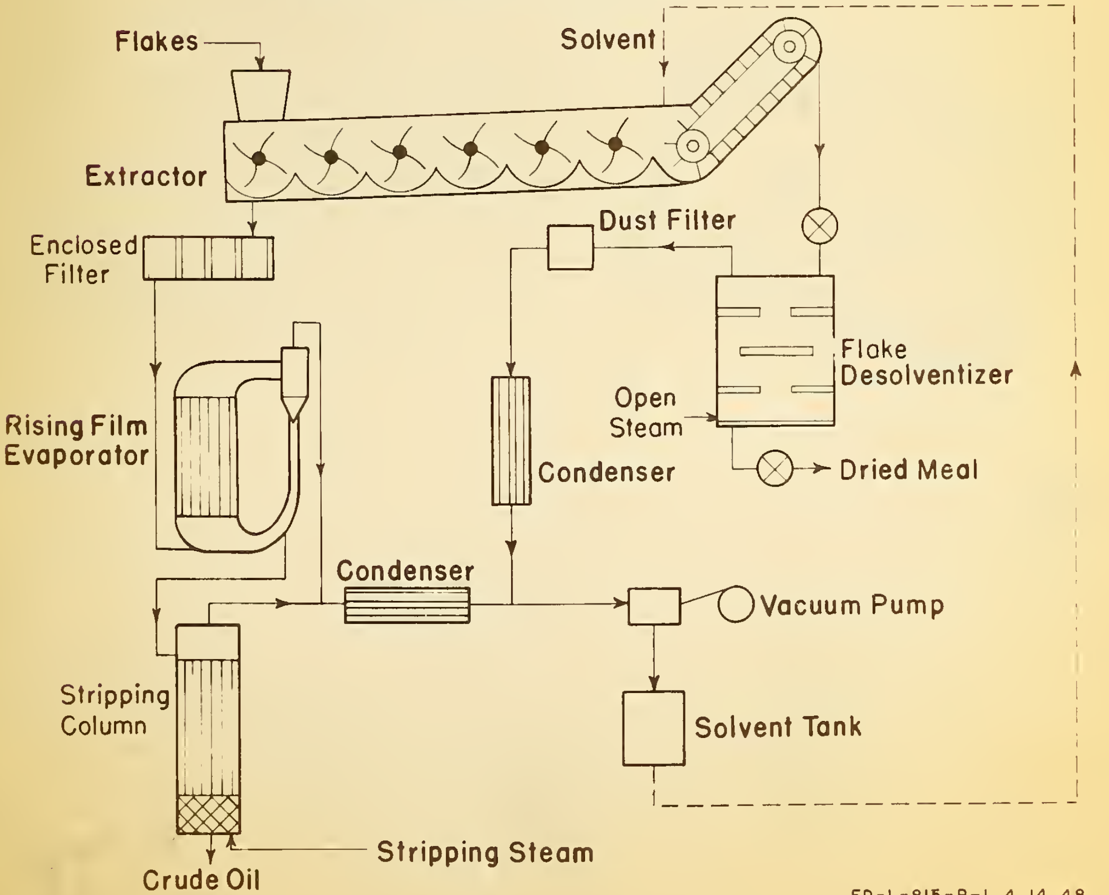
FIG. 2.– OILSEED EXTRACTION



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